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Technical Report 32-1237

Isotropic Plasticity of Graphite

D. B. Fischbach

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JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY
PASADENA, CALIFORNIA

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Isotropic Plasticity of Graphite

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Abstract

A study of the dimensional changes accompanying the high temperature plastic deformation of a pyrolytic carbon suggests that this material deforms like a homogeneous, isotropic solid. At the end of the first stage of deformation a width increase of about 3% and a thickness decrease of about 12% had occurred. However, upon continued tensile elongation both the width and the thickness were observed to decrease at the same rate as a function of elongation.

Isotropic Plasticity of Graphite

I. Introduction

The anisotropy of graphite is one of its best known features. Because of the strongly anisotropic layers of its crystal structure, most transport, electronic, and mechanical properties have very different values when measured parallel to the layer (basal) planes than they have when measured perpendicular to these planes. However, some observations by Kotlensky (Ref. 1) on dimensional changes accompanying tensile deformation of pyrolytic carbons suggest that this may not be so for high temperature plastic deformation. Recently, more precise data on the tensile deformation of highly oriented, well-graphitized pyrolytic carbon have confirmed and extended Kotlensky's results. It appears that this material deforms like a homogeneous, isotropic solid.

II. Materials and Procedures

Dog-bone-type specimens of as-deposited pyrolytic carbon were elongated in tensile creep parallel to the substrate plane at temperatures of 2500–2900°C and stresses of 5,000–30,000 lb/in^2. The uniform gage-section elongation was determined from measurements at room temperature of the change in distance between fiducial marks drawn on the specimen with black acetate drawing ink. Such ink lines, placed on the specimen before testing, were still clearly visible after extensive deformation at temperatures as high as 2900°C. The width (parallel to the average layer plane orientation) and thickness (normal to the layer planes) at the center of the gage section was also measured with a micrometer before and after deformation. Some measurements were also made on specimens deformed at constant cross-head rate in a tensile machine. These results are shown in Fig. 1.

Pyrolytic carbon was deposited at 2200°C from methane by a commercial producer[1] and had the usual substrate-nucleated growth-cone microstructure. The initial stage of deformation (dashed lines, Fig. 1) is characterized by dewrinkling (straightening) of the as-deposited *wrinkled sheet* microstructure. Although this process proceeds more rapidly and completely in the stress direction, substantial dewrinkling normal to the stress axis also accompanies tensile deformation. This results in an increase in gage-section width and a rapid decrease in gage-section

[1]Super Temp Corp., Santa Fe Springs, Calif.

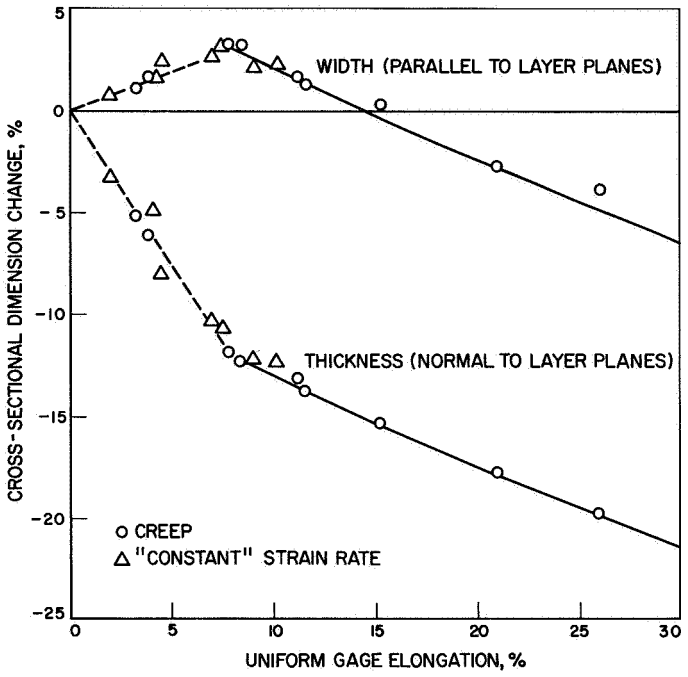

Fig. 1. The change in cross-sectional dimensions of pyrolytic carbon as a function of tensile elongation parallel to the substrate. The solid curves were calculated assuming isotropic constant-volume deformation for elongations $\geq$ 8%

thickness (Refs. 1, 2). At the end of this first stage of deformation (about 8% elongation for the particular carbon used here) an increase in width of about 3% and a decrease in thickness of about 12% had occurred. The growth-cone microstructure had completely disappeared on the gage-section edges (parallel to stress, normal to substrate) but could still be detected in cross sections (normal to stress and substrate); and x-ray diffraction preferred orientation measurements showed that the average layer plane orientation lay within 2 degrees or less of the stress axis (Ref. 2). At this stage, the layer plane orientation texture could be described as weakly corrugated parallel to the stress direction. The material was also well graphitized with an interlayer spacing $d \leq 3.36$ Å.

With continued tensile elongation of this highly oriented and well-graphitized material, both the width (parallel to layer planes) and the thickness (normal to layer planes) decreased. Both dimensions decreased at the same rate, within the error of these experiments, as a function of elongation! The solid lines in Fig. 1 were calculated on the assumption of constant-volume isotropic deformation for elongations $\geq$ 8%. Agreement with the data is excellent. Carbon evaporated from the heating element tended to deposit on the specimen layer plane edges, but not on the layer plane faces, a fact which may account for the somewhat greater scatter in the width results. The assumption of constant-volume deformation was verified by the reduction in cross-sectional area that was calculated from the width and thickness measurements. During the first stage of deformation the reduction in area increased with elongation more rapidly than the theoretical constant-volume rate. After about 8% elongation the observed reduction in area was greater than the calculated value by about 2%. Thereafter, the deformation occurred at constant volume within the precision of the data. The 2% excess in reduction in area corresponds closely to the decrease in interlayer spacing (from about 3.42 Å to $\leq$ 3.36 Å) which accompanies the graphitization. Microstructural examination of specimens elongated as much as 34% gave no evidence of twinning or kinking in the gage section. Delamination cracks were prevalent, but the reduction in area results indicates that these may have resulted from preparation of the metallographic specimen rather than from the high temperature deformation.

III. Results

The results in Fig. 1 show that tensile creep deformation of highly oriented, well-graphitized pyrolytic carbon parallel to the layer planes occurs isotropically and at constant volume. No conclusive explanation can be given for these observations at present. However, this behavior appears to have relevance to the question of deformation and self-diffusion mechanisms in pyrolytic carbon, and perhaps in other forms of graphite as well. First of all, it is clear that deformation mechanisms other than basal shear, or kinking and twinning, do operate in graphite at elevated temperatures. Whatever the deformation mechanisms are that are operating, they must provide approximately equal deformation components normal and parallel to the c-axis. For example, if non-basal dislocation glide is the dominant mechanism, slip on prism planes alone will not suffice; pyramidal glide with a slip vector component parallel to the c-axis must also be present. At present, there is little direct evidence to support glissile non-basal dislocations. On the other hand, Nabarro-Herring mass-transport diffusion may be the dominant deformation mechanism, as suggested by observations on the stress-dependence of the creep rate (Ref. 3). In that case, significant mass transport must occur in the c-axis direction as well as parallel to the

layer planes. It is difficult to assess the relative diffusion rates on this basis; they should depend on the reciprocal squares of the diffusion distances in each direction, which are not known. If it is assumed that crystallite boundaries are the active sources and sinks of the diffusing defects, that the crystallite diameter/thickness ratio is ≤ 2 (Ref. 4), and that layer plane diffusion is the rate-determining process, then c-axis diffusion may be less than an order of magnitude slower than layer plane diffusion. On the other hand, if the crystallite diameter/thickness ratio were ≥ 10 (Ref. 5), then c-axis diffusion could be several orders of magnitude slower than layer plane diffusion and it should then be the rate-controlling process. The observed creep activation energy in both stages of deformation was 250 ± 30 kcal/mole, which is an appropriate value for diffusion by a vacancy mechanism in the layer planes (Refs. 2, 6). It is difficult to conceive of a c-axis diffusion mechanism that does not involve interstitial atoms. One exception is vacancy diffusion along c-axis screw dislocations or similar defects. In macroscopic observations such as the present dimension change data on polycrystalline material, such short-circuit paths could be responsible for apparently near-isotropic diffusion rates. However, this restrictive condition may not be necessary. Thrower (Ref. 7) has found evidence for substantial interstitial c-axis diffusion in electron microscope studies of the kinetics of annealing of high temperature radiation damage in single crystal and well-annealed pyrolytic graphites. It might be argued that vacancy diffusion is more likely than interstitial diffusion in unirradiated material, because the formation energy of a vacancy is believed to be appreciably lower than the formation energy of an interstitial defect, and the creep activation energy is that expected for vacancy diffusion. However, Thrower has implied that the diffusion activation energy (the sum of the defect formation and migration energies) may be approximately the same for interstitial diffusion parallel to the c-axis and vacancy diffusion normal to the c-axis. This is consistent with the present results.

IV. Conclusion

Clearly, much remains to be learned about both diffusion and deformation mechanisms in graphite. However, the present results support a conclusion already reached by Thrower: although the assumption has frequently been made that graphite is essentially a two-dimensional material, this simplification may not be realistic when high temperature diffusion or plastic deformation processes are being studied. The three-dimensionality of graphite has also been confirmed in the electronic behavior, especially the diamagnetic susceptibility (Refs. 8, 9).

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